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Certification

Park IP Translations

This is to certify that the attached translation is, to the best of my knowledge and belief, a true and accurate translation from Japanese into English of: Japan Patent Office Published Unexamined Patent Application No. S53-134759, Publication Date: November 24, 1978 and entitled: "Method for Manufacturing Metal Composite Powder."

A handwritten signature in cursive script, reading 'Abraham I. Holczer', is written above a horizontal line.

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(54) Method for Manufacturing Metal Composite Powder

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Specification

1. Title of the Invention

Method for Manufacturing Metal Composite Powder

2. Claims

A method for manufacturing a metal composite powder that uses a silver complex salt solution, the main components of which are the three components of silver nitrate, ammonium carbonate salt, and ethylenediamine-4-acetic acid-3-sodium salt, and electrolytically replaces a metallic copper powder surface with metallic silver.

3. Detailed Description of the Invention

The present invention relates to a method for manufacturing a silver coated metal composite powder excellent for use as a conductive filler. It is an object thereof to provide a metal composite powder capable of imparting conductivity to the same extent as silver particles, when dispersed in a synthetic resin binder instead of silver particles as a powdered conductive filler, to a coating constituted of the same.

Conventionally, a metal powder, carbon powder, or the like powdered conductive filler is dispersed in a synthetic resin binder, is for example made into a conductive coating material, and after application, is known to impart conductivity to a coating. As a conductive filler, carbon powder has an inexpensive price and stability over time; however, the electrical resistance is high after formation of the coating material at 10^{-1} Ω -cm or greater, and it may be used as a resistive element, but is unsuitable for an electrical conductor. The electrical resistance of silver particles is low at 1.62×10^{-6} Ω -cm for metallic silver and close to 10^{-4} - 10^{-5} Ω -cm after forming a coating material, also having the advantages of oxidation resistance and stability over time. However, the price is high for an industrial material, and moreover there are problems in terms of resources thereof. From this point of view, powders where silver is deposited on the surface of powders such as conventional carbon, silicon oxide, nickel, copper and the like are being developed as composite powders; however, of these, the resistance values for ones with silver deposited on the surface of carbon powder and silicon oxide powder are high. The particle size is also a large, and formation of a coating material is difficult. Moreover, there are the disadvantages of the adherence of the base material powder and the silver coating being insufficient and stability being lacking. In addition, to impart excellent conductivity to powders in which silver is deposited on the surface of a nickel powder, silver content of 60% or greater is necessary; therefore, the effect on price reduction is small.

In addition, in powders where silver is deposited on the surface of a copper powder, the silver is deposited on the surface of the copper from a silver cyanide by displacement plating as proposed, for example, in Published Examined Patent Application No. S47-3019. As described in the specification thereof, silver cyanide is used as a base substance in a plating solution, and to make the silver cyanide present in this plating solution, an alkali metal cyanide (soda *[typographical error in source, not translatable]* cyanide) is used in a high concentration. Therefore, there are the disadvantages not only of an extremely high danger of toxicity in working environments, but also of poor adhesion of the deposited silver to the copper and the silver coating easily peeling in mechanical mixing processes when being dispersed in the synthetic resin binder, as well as increases in the resistance value because of oxidation of the copper bus exposed and a lack of stability over time. Problems that must be improved are present.

Moreover, as is described in the specification of Published Examined Patent Application No. S47-3019 above, the reality is not being able to obtain a good electrically conductive silver coating by reduction plating using negative ions other than cyanides.

The present invention has been originated as a solution in light of the problems described above, and provides a method for manufacturing an excellent silver coated metal composite powder having superior adhesion for copper powder and a silver coating, having conductivity substantially the same as silver particles as a conductive filler, and being inexpensive without any danger of toxicity in the work environment during manufacturing.

Next, the constitution of the present invention will be described. A silver complex salt solution of which the main components are the three components of silver nitrate, ammonium carbonate salt, and ethylenediamine-4-acetic acid-3-sodium salt (hereinafter called EDTA) is used, and the silver coated metal composite powder is obtained by electrolytic replacement of a metallic copper powder surface with metallic silver. Moreover, the ammonium carbonate salt of the present invention is ammonium carbonate and ammonium bicarbonate. Thus, the compositional ratio that may be used for the silver complex salt solution for forming a silver coating having excellent adherence on the surface of the copper powder in the present invention is in the range of 1 - 40% (by weight) silver nitrate, 10 - 80% (by weight) of the ammonium carbonate salt, and 20 - 80% (by weight) EDTA.

What limited the compositional ratio of the silver complex salt solution as described above is not obtaining a silver coating of satisfactory thickness for the composite powder if the silver nitrate is less than 1% (by weight) and, on the other hand, deposition of a uniform silver coating becoming difficult and yield being reduced if the insoluble components in the silver complex salt solution come to exceed 40% (by weight). If the ammonium carbonate salt is less than 10% (by weight) or greater than 80% (by weight), the adhesion of the silver coating tends to become worse. If the EDTA is less than 20% (by weight) or greater than 80% (by weight), the adhesion of the silver coating becomes poor, and at the same time, if the same is less than 20% (by weight), the electrical resistance of the coating tends to increase after the formation of the coating material using the composite powder obtained.

From the reasons above, such as solubility in the silver complex salt solution, adhesion of the silver coating, and electrical resistance of the composite powder obtained, a range of 3 - 30% (by weight) silver nitrate, 15 - 70% (by weight) ammonium carbonate salt and 23 - 75% (by weight) EDTA may be used preferably, and in particular, a range of 5 - 20% (by weight) silver nitrate, 25 - 55% (by weight) ammonium carbonate salt, and 25 - 70% (by weight) EDTA most preferably.

The concentration of the silver complex salt solution used in the present invention is not limited in particular, and a wide range from a low concentration to a high concentration may be used, but from the solubility of the various components of the silver complex salt solution, a concentration of 5 - 40% (by weight) is normally used.

To manufacture the silver coating composite powder in the present invention, the same is obtained just by mixing and stirring the previously described silver complex salt solution and copper powder. The reaction temperature during mixing may be normal temperature, or there may be heating. 20 - 60°C is preferable because the reaction rate is increased.

The proportion of the composite powder copper and silver in the present invention is not limited in particular, but if there is too little silver, a silver coating with a sufficient thickness will not be obtained; therefore, for example, the copper may be exposed by abrasion of the silver coating in mechanical kneading during the formation of the coating material. On the other hand, as the silver increases, the price becomes higher, so it is not preferable. The preferable proportion when represented in percent by weight is 50 - 95 of copper to 5 - 50 of silver, more preferably 7 - 40 silver: 60 - 93 copper, and most preferably 10 - 30 silver: 70 - 90 copper.

Moreover, the metallic copper powder used in the present invention may be produced by any method such as replacement methods that make use of electrolysis and ionization tendencies, atomizing methods, mechanical pulverization, and the like. In addition, the shape of the copper powder may be any of spherical, flat shaped, resin shaped, and needle shaped, and the size is not limited in particular.

Next, the description will be further supplemented by giving examples of the present invention.

Example 1

1000 g of an approximately 5% aqueous solution of H_2SO_4 is added to 1000 g of Cu powder (electrolytic copper powder from a No. 325 mesh or smaller), and acid cleaning is carried out by stirring at 20°C for 5 - 10 minutes, dissolving and eliminating the copper oxide surface layer. Next, warm water at 25 - 35°C is added to the Cu powder, and washing is repeated until the filtrate becomes neutral. This time, the number of washings was six, and 30ℓ of washing water were used.

Next, the wet Cu powder described above was transferred to an approximately 15 ℓ polyethylene container, and a silver salt solution with the following composition was added. When stirring was continued for approximately 60 minutes at a stirring speed of approximately 200 rpm at a temperature of 20 - 25°C, a silver coated metal composite powder was obtained.

| | | | |
|----------------|---------|--------------|--|
| $AgNO_3$ | 375 g | | |
| H_2O | 750 cc | } Solution A | |
| $(NH_4)_2CO_3$ | 1875 g | | |
| NDTA | 1500 g | | |
| H_2O | 8000 cc | } Solution B | |

(Here, solution A is gently added after solution B has dissolved and become transparent. Used after the precipitate, which temporarily arises because of the addition, has dissolved.)

The metal composite powder produced is repeatedly washed until the filtrate becomes colorless at a temperature of 25 - 35°C, and thereafter, the product is produced by drawing at 70 - 100°C. The theoretical values for replacement and the measured values in this case are as follows.

| | | Gram equivalency | Equivalency |
|-----------|--------|------------------|-------------|
| Cu powder | 1000 g | 65/2 | 30.77 |
| $AgNO_3$ | 375g | 170 | 2.21 |

| | Theoretical value | Measured value |
|------------------------------|-------------------|----------------------|
| Ag content in Cu powder | 20.45% | 20.60% |
| Metal composite powder yield | 1167 g | 1155 g (99.0% yield) |

To test the conductivity of the metal composite powder produced as per the above, the following composition was kneaded for two hours in a ball mill and applied.

| | |
|---|------|
| Metal composite powder | 90 g |
| Methyl methacrylate polymer solution (30% toluene solution) | 33 g |
| Toluene / butyl acetate (1/1) | 10 g |

Using the conductive coating material above, application on a phenol resin sheet was such that the dry coating thickness was 1.5 microns, and drying was carried out for 30 minutes at 100°C. The specific resistance of this coating was $2 \times 10^3 \Omega \cdot cm$, and excellent conductivity was exhibited.

In addition, a 1.5 mm (φ) copper wire was used to test the adherence of the silver coating to copper, and a silver coated copper wire was produced by acid washing and carrying out silver replacement with the same conditions as before. When this was repeatedly bent at a right angle with bending with a radius of 10 mm, no cracks, peeling, or other defects arose in the silver coating even when bent 10 times. Extremely good adhesion was exhibited.

Moreover, to test the superiority of the adhesion of the silver coating of the present invention, a silver cyanide replacement plating solution with the following composition was used, and a silver coated copper wire was produced in the same manner as before. When the same bending tests were carried out, the silver coating cracked and a peeling phenomenon arose with being bent only three times.

| | |
|----------|--------|
| $AgNO_3$ | 170 g |
| NaCN | 196 g |
| H_2O | 3294 g |

Examples 2 - 11 and comparative examples 1 and 2

Silver complex salt solutions with each of the compositions in Table-1 below were created. (The results for the solubility thereof are given in Table-1.) Using these silver complex salt solutions, composite powders and silver coated copper wires were produced by the identical method as in Example 1. The coating material was formed with composite powders obtained by methods identical to Example 1, and the specific resistances of dried coatings for each are given in Table-1. In addition, silver coating adherence tests the same as Example 1 were carried out for the silver coated copper wires, and the results for these are also given in Table-1.

Table 1

| | Examples | | | | | | | | | | Comparative Examples | |
|---|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|----------------------|-----|
| | | | | | | | | | | | 1 | |
| AgNO ₃ | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 20 | 20 | 285 | 50 | 16 |
| (NH ₄) ₂ CO ₃ (g) | 70 | 60 | 40 | 30 | 20 | 10 | - | 40 | - | 38 | 30 | 75 |
| (NH ₄)HCO ₃ (g) | - | - | - | - | - | - | 40 | - | 40 | - | | |
| EDTA (g) | 20 | 30 | 50 | 60 | 70 | 80 | 50 | 40 | 40 | 33.5 | 20 | 9 |
| H ₂ O (g) | 230 | 230 | 230 | 230 | 230 | 230 | 230 | 230 | 230 | 230 | 230 | 230 |
| (Test results) (Notes) | | | | | | | | | | | | |
| Silver complex salt solution solubility | ○ | ○ | ○ | ○ | ○ | Δ | ○ | Δ | Δ | (x) | x | Δ |
| Specific resistance Ω-cm | 2×10 ⁻¹ | 7×10 ⁻² | 4×10 ⁻³ | 5×10 ⁻³ | 2×10 ⁻³ | 3×10 ⁻³ | 3×10 ⁻³ | 8×10 ⁻³ | 6×10 ⁻³ | 1×10 ⁻² | - | - |
| Adherence | Δ | ○ | ○ | ○ | Δ | Δ | ○ | ○ | ○ | ○ | - | x |

(Notes) Symbols for test results

Silver complex salt solution solubility

○: Dissolved and transparent

Δ: Small amount of insoluble components present

○: Took time to dissolve and become transparent

x: Large amount of insoluble components

Adhesion

○: No defects arising with 10 bending tests

Δ: Small amounts of cracking occurs with 5- 10 bendings

x: Cracking occurs with one bending

As is clear from the results in Table-1, if the composition of the silver complex salt solution is within the limited range described above, as is shown by Examples 2 - 11, a composite powder can easily be produced. The specific resistance of a coating of a coating material using the composite powder obtained was also a value that does not make for any practical impediment, and the adherence of the silver coating to copper was excellent. Conversely, the [composite powder] of Comparative Example 1 that had AgNO₃ in the composition of the silver complex salt solution exceeding the limited range had a large amount of insoluble components, and it was difficult to produce a composite powder. In addition, the one in Comparative Example 2 in which EDTA outside of the range below the limit amount had poor adhesion of the silver coating and could not stand up to use

The metal the composite powder produced by the present invention not only has extremely superior electrical conductivity as a conductive filler, but also provides various effects such as the adhesiveness thereof being superior and avoiding the danger of toxicity in the work environment.

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⑭金属複合粉の製造法

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明 細 書

1. 発明の名称

金属複合粉の製造法

2. 発明技術の範囲

導電紙、炭様アンモニウム塩およびエチレンジアイン4部、酸3ナトリウム塩の三成分を主成分とする銅塩溶液を用いて金属銅粉の表面に金属銅を塩析析出させることを特徴とする金属複合粉の製造法。

3. 発明の詳細な説明

この発明は、導電フィルターとして用いられる改良された銅被覆金属複合粉の製造法に関するものであり、粉状の導電フィルターとして銀粒子の代りに合成樹脂バインダー中に分散させ、導電性塗料として使用した場合それにより構成される塗膜に、銅粒子と同程度の導電性を付与することである金属複合粉を提供することを目的とするものである。

従来、金属粉あるいはカーボン粉などの粉状の導電フィラーを、合成樹脂バインダー中に分散さ

せて例えば導電性塗料とし、塗布後その塗膜に導電性を付与することは知られている。導電フィルターとしてのカーボン粉は価格が安く導電性にも安定しているが、電気抵抗が塗料に換算して $10^{-1} \sim 10^{-2} \Omega \cdot \text{cm}$ 以上と大きく、抵抗体としては使用し得るが導体としては不満足である。銀粒子は電気抵抗が金同様に $1.62 \times 10^{-8} \Omega \cdot \text{cm}$ 、塗料に換算して $10^{-4} \sim 10^{-5} \Omega \cdot \text{cm}$ と小さく、かつ耐酸化性で経時的にも安定しているという長所はあるが、工業材料としては価格が高く、その上資源的な問題がある。類かる銅系から、従来カーボン、酸化亜鉛、ニッケル、銅等の粉末の表面に銅を析出させたものが導電粉として用いられているが、このうちカーボン粉、酸化亜鉛粉の表面に銅を析出させたものは電気抵抗が大で、粒度も大きく、塗料化が困難であり、その上基材粉末と銅被覆との密着性が充分でなく安定性に欠ける欠点がある。又、ニッケル粉の表面に銅を析出させたものは、良好な導電性を付与するのには $\text{Ag content } 60\%$ 以上必要とするので低価格下に及ぼす効果は小さく、又銅粉の表面に銅を析出させ

たものは、例えば特許第 47 - 5019 号の明細書にみられる如く、金属メッキ液によって銀シアン化物より銅表面に膜を析出させるものであるとして、その明細書に説明されているように金属前液にシアン化物が主体として用いられ、この金属前液中にシアン化銀を存在させるためにアンモニア金属シアン化物（シアン化ナトリウム）が高濃度で使用されている。したがって、作業環境における毒性の危険が極めて高いばかりでなく、析出した銀の腐に列する腐食性に乏しく合成樹脂（バインダー）中に分散せしめる際の機械的剥離工程で銀被膜が剥離し易く、このため露出した銅の酸化による低電位の増大が生じて経時腐食に欠けるという欠点があり、改善しなければならぬ問題が存在していた。

尚、前記特許第 47 - 5019 号の明細書にも記載されているように、シアン化物以外の銅の錯イオンを配用する還元メッキ法でも良好な導電性の銀被膜が得られないのが現状であった。

この発明は、上述の問題に鑑みこれを解決するために創案されたものであるとして、銅粉と銀被膜の

(a)

（重量）を多量に多くするにつれて銀被膜溶解に不溶性成分が多くなり、均質な銀被膜の析出が困難になるばかりでなく、収率も低下することによる。銀被アンモニウム塩は 10（重量）よりも少なくても、又 80（重量）よりも多くても銀被膜の密着性が悪くなる傾向があることによる。また、銅粉が 20（重量）よりも少なく、又 80（重量）よりも多くなると銀被膜の密着性が悪くなるのと同時に 20（重量）よりも少なくなると得られた被膜面を用いて塗料化した際の塗膜の密着性が低下する傾向があることによる。

以上のような銀被膜溶解の経路性、銀被膜の密着性および得られた被膜面の電抵抗値等の理由から、銅被膜は 5 - 50（重量）%、銀被アンモニウム塩 15 - 70（重量）%、および銅粉 15 - 25（重量）% の範囲が好ましく、銅被膜 5 - 20（重量）%、銀被アンモニウム塩 25 - 55（重量）%、および銅粉 15 - 25（重量）% の範囲が最も好ましく使用可能である。

この発明で用いる銀被膜溶解の濃度は、特に銀

特許第 53 - 13478 号

面密性が流れ、導電フィラーとして銀粒子とほぼ同等の導電性を有し、安定かつ高速時の作業環境に適合の危険の全くない、改良された被膜被覆液の製造法を提供するものである。

次に、この発明の構成について説明すると、銀被膜、銀被アンモニウム塩および銅被アンモニウム塩のナトリウム塩（以下それぞれという）の三成分を主成分とする銀被膜溶解液を用いて金属表面に金属被膜を生成析出させることにより銀被膜金属被覆液を得るものである。なお、この発明でいう銀被アンモニウム塩とは銀被アンモニウムおよび銀被アンモニウムである。しかして、この発明において銅粉の表面に良好な密着性を有する被膜を形成せしめるための銅被膜溶解液の組成比は銅被膜 1 - 40（重量）%、銀被アンモニウム塩 10 - 80（重量）%、および銅粉 20 - 60（重量）% の範囲で使用可能である。

銀被膜溶解液の組成比を前述のように限定したのには、銅被膜は 1（重量）% より少くなると被膜として満足な厚さの銀被膜が得られず、一方 40

(b)

であるものではなく、低濃度から高濃度までの広範囲で使用できるが、銀被膜溶解液の各成分の溶解特性から通常 5 - 40（重量）% 濃度で使用される。

この発明における銀被膜被覆液を製造するには、前述の銀被膜溶解液と銅粉を混合溶解するだけで得られる。混合時の反応温度は常温でもよく、また加熱してもよい。20 - 60℃ の範囲は反応速度を早める上で好ましい。

この発明における被覆液の銅と銀の比率は、特に銅被膜はよいが、銅が少な過ぎると充分な厚さの銀被膜が得られないから、銅は塩析化の後の機械的な剥離工程で銀被膜が剥離して銅が露出することがあり、一方、銅が多くなると銅粉が高くなるので好ましくない。至極まで狭い被膜の好ましい比率は、銅 5 - 50 に対して銅 50 - 95 であり、より好ましくは銅 7 - 40；銅 60 - 95、最も好ましくは銅 10 - 30；銅 70 - 90 である。

尚、この発明に用いる金属被覆液は、銅粉、イオン化銅粉を用いた電鍍液、フッ素系、有機溶剤、銅粉等を用いた方法で造ってもよい。また銅

(c)

-288-

(d)

粉の形状も球状、扁平状、樹脂状、針状等のいずれでもよく、粉末の大きさも特に限定しない。

次に、この発明の実施例を示して更に説明を補足する。

実施例 1:

Cu 粉末 (325 ノンシム以下の電解銅粉) 1000g に約 5 分の H₂SO₄ 水溶液 1000g を加え、20℃/5-10 分浸漬して酸化を行い、表面の酸化銅を溶解除去する。次いで該 Cu 粉末を 25-35℃の硝酸を加えて硝酸が中性になるまで洗滌を繰返す。この時の洗滌回数 6 回、洗滌水は 50ℓ 使用した。

次に、上記の濯洗している Cu 粉末を約 15ℓ のポリエチレン製容器に移し、下記組成の銀塩塩溶液を加えた。恒温 20-25℃、浸漬温度約 20℃、pH で約 60 分浸漬を続けて乾燥せしめたところ、銀塩合金粉末が得られた。

AgNO₃ 375g
H₂O 7500g) 溶液

例

以上のようにして調製した金属複合粉の導電性を減衰するため、下記組成のものをボールミルで 2 時間混練して塗布化した。

金属複合粉 90g
メタメルトリレート重合体溶液 (30wt% 溶液) 35g
トリエノリ樹脂アグレス (1/1) 10g

上記導電性塗料を用いて、フェノール樹脂板に乾膜厚が 15 ミクロンになるように塗布し、100℃で 50 分乾燥した。この塗膜の比抵抗は $2 \times 10^{-3} \Omega \cdot \text{cm}$ であり、良好な導電性を示した。

また、銅に対する銀塩の溶解性を試験するため 1.5mm 径の銅板を用い、前記と同一条件で乾燥、銀置換を行って銀被覆銅板を製造し、これを径 10mm の円形で直角に折ることを繰返したところ 10 回の折曲げによっても銀被覆面に亀裂あるいは剥離等の欠陥が現れず、極めて良好な耐腐性を示した。

なお、この発明の銀塩の溶解度の優位性を試験するため、下記組成のシアン化銀溶液を用いた。

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(NH₄)₂CO₃ 1875g
NH₄TA 1500g } B 液
H₂O 8000g

(但し、B 液が溶解透明化してから A 液を緩かに添加する。添加により一時的に生じた沈殿が溶解した後用いる)

乾燥した金属複合粉は 25-55℃の温度で溶液が黒色になるまで洗滌を繰返し、しかるのち分別し、70-100℃で乾燥して製品とした。この場合の乾燥の理論値と実測値は次の如くであった。

| | | | |
|--------------------|-------|-------|-------|
| | | 瓦 量 量 | 備 註 |
| Cu 粉末 | 1000g | 65/2 | 50.77 |
| Ag NO ₃ | 375g | 170 | 2.21 |

| | 理論値 | 実測値 |
|----------------|--------|------------------|
| Cu 粉末中の Ag 含有率 | 20.45% | 20.0% |
| 金属複合粉収量 | 1167g | 1155g (収率 99.0%) |

例

を用いて前記同様にして銀被覆銅板を製造し、同様に折曲げ試験したところ、5 回の折曲げによつて屈曲部の被覆面に亀裂および剥離現象が生じた。

AgNO₃ 170g
NaCN 196g
H₂O 5294g

実施例 2-11. および比較例 1.2

下記表-1 の組成の銀塩塩溶液をそれぞれ作成した。(その溶解性についての結果を表-1 に示した)。この銀塩塩溶液を用いて実施例 1 と同一方法で複合粉および銀被覆銅板を製造した。得られた複合粉について実施例 1 と同一方法で塗料化した、それぞれの乾燥後の比抵抗を表-1 に示した。また銀被覆銅板について、実施例 1 と同様に銀被覆の耐腐性を試験し、その結果を合せて表-1 に示した。

表 1

| 成分 | 天 | | | | 地 | | | | 水 | | | | 正 規 質 | | | |
|--|------|------|------|------|------|------|------|------|------|------|------|------|-------|------|------|------|
| | 10 | 15 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 |
| Ag ₂ PO ₃ (g) | 10 | 15 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 |
| [H ₂], O ₂ (g) | 70 | 80 | 40 | 30 | 20 | 10 | — | — | — | — | — | — | — | — | — | — |
| [NH ₄], O ₂ (g) | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — |
| 2.5 T.A. (g) | 20 | 30 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 |
| H ₂ O (g) | 200 | 220 | 220 | 220 | 220 | 220 | 220 | 220 | 220 | 220 | 220 | 220 | 220 | 220 | 220 | 220 |
| [正 規 質 1 部] | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — |
| 製造業者の保証書 | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — |
| 出 産 年 月 日 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 |
| 製 造 年 月 日 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 |
| 製 造 年 月 日 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 | 2012 |

注) 試験結果の記号

製造業者の保証書

○: 製造業者

◎: 製造業者の不適合品あり

○: 製造業者の不適合品あり

x: 不適合品あり

製 造 年

○: 製造業者の保証書

◎: 製造業者の不適合品あり

○: 製造業者の不適合品あり

x: 不適合品あり

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表 1 の結果から明らかとなり、製造業者の保証書が実際の製造 2 ～ 11 で示したように前記保証書範囲内であれば適合品が容易に製造でき、得られた適合品を用いた試験結果の比較値も実用上何ら支障のなす所であり、更に列する製造業者の製造性も良好であった。これに対し製造業者の保証書中 Ag₂PO₃ が製造範囲を超えて多い比較例 1 のものは不適合品が多く、適合品の製造が困難であり、また H₂O が製造範囲を超えて少ない比較例 2 のものは製造業者の製造性が悪く使用に耐えないうのであった。

この結果によつて製造された全製造適合品は、導電ファイバーとして極めて導電性に優れているばかりでなく、その製造性も優れており、かつ作製強度がその他の危険から避けられるなどの諸効果をもたらすものである。

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